

358

**ADVANCED
TECHNOLOGY
LABORATORIES**

N64-28962

(ACCESSION NUMBER)

35

(PAGES)

CR-58539

(NASA CR OR TMX OR AD NUMBER)

(THRU)

(CODE)

17 (CATEGORY)

**PROGRESS
REPORT**

NO. 7

R

**DEVELOPMENT OF LOW TEMPERATURE
DIELECTRIC COATINGS FOR
ELECTRICAL CONDUCTORS**

BY
K.N. MATHES

APRIL 15, 1963

Seventh Quarterly Report
April 15, 1963

Development of Low Temperature Dielectric Coatings
for Electrical Conductors

Contract No. ~~NAS8-2442~~
Control Number: TP85-498(CPB-02-1096-61)
(W/O #8500-0-0224-000-8500-11-431)

General Electric Company
Missile and Space Vehicle Department
Philadelphia, Pa.

Report Prepared by:

K. N. Mathes
Advanced Technology Laboratories
General Electric Company
Schenectady 5, N. Y.

Report Prepared for:

George C. Marshall Space Flight Center
Huntsville, Alabama

Seventh - Quarterly Report
April 15, 1963

DEVELOPMENT OF LOW TEMPERATURE DIELECTRIC COATINGS
FOR
ELECTRICAL CONDUCTORS

INTRODUCTION

The continuing evaluation of the effect of thermal aging on the performance of wire insulation at room and cryogenic temperatures constitutes the major part of the work described in this report. The procedures and test facilities were described in the fifth quarterly report dated October 15, 1962 and the first part of the work (which is continued in this report) is described in the sixth quarterly report dated January 15, 1963. In this report, mandrel flexibility and dielectric measurements are summarized for all of the wires after aging the specified length of time in vacuum and air at 120 C and in air at 250 C. The results reported go considerably beyond the contract responsibilities in that after aging measurements have been made in liquid helium and sometimes in liquid nitrogen even though not specifically required.

Data is given also for wires after exposure to high humidity but these data are not yet complete. Information on the design of test facilities for this and other requirements has been included also.

This report describes data on the flexibility of several ribbon cable constructions while immersed in liquid helium.

The dielectric evaluation of cryogenic liquids has been plagued by bad luck but the newly developed test equipment is described.

SUMMARY AND CONCLUSIONS

Wire Evaluation

Thermal Aging

It is gratifying to report that the effects of thermal aging in air and vacuum on wire insulation can be determined by measuring changes in flexibility, voltage breakdown and dissipation factor and that correlation between these different methods can be shown at least qualitatively. Each measurement approach has advantages and disadvantages which differ with the various wire insulations. Measurement of flexibility at cryogenic temperatures is often a particularly sensitive tool for following

the degradation caused by thermal aging. However, plasticized poly-vinyl chloride (PVC) is so brittle at cryogenic temperatures that such measurements are useless as a tool for following the effects of thermal aging. Unfortunately, changes in flexibility at room temperature do not measure changes caused by thermal aging in PVC either. While voltage breakdown at room temperature is not sensitive to thermal aging changes in PVC, similar breakdown measurements made at cryogenic temperatures do indicate the progressive degradation of thermal aging in vacuum (see Fig. 7).

It is important to find the most appropriate and suitably sensitive test technique for determining degradation in each type of material. Flexibility and voltage breakdown* measurements at cryogenic temperatures have been shown to be particularly sensitive and more suitable than the room temperature measurements in following progressive thermal degradation. It follows that thermal aging must also be a functionally important parameter when considering the use of insulating materials exposed in application to both high and low temperatures.

For example, PVC even before aging is brittle at cryogenic temperatures but if undisturbed maintains high voltage breakdown. When PVC is thermally aged in vacuum, the voltage breakdown at cryogenic temperatures is significantly degraded. It is important to note that with PVC the aging in vacuum at 120 C produces significant degradation while aging in air did not. Precisely the reverse is true with Formex, which exhibits significant degradation when aged in air at 120 C but not in vacuum! Obviously the mechanism of degradation is different for the two types of materials. With PVC, degradation is apparently produced by loss of volatiles (such as plasticizers) in vacuum while for Formex, degradation appears to be related to cross linking and embrittlement produced by oxidation in air.

For ML, degradation with aging appears to be insignificant at 120 C but at 250 C in 120 days appears to occur primarily as a result of copper oxidation which causes loss of adhesion detected by flexibility tests at both room and cryogenic temperatures. However, the initial cure and the development of the optimum molecular structure in ML does influence the flexibility at cryogenic temperatures which thereby becomes a useful evaluation tool. It is likely that cryogenic flexibility will detect changes caused in the molecular structure of ML with sufficient thermal aging but 120 days at 250 C is not enough to cause significant change in this very thermally stable material.

The changes in dissipation factor of thermally aged wires are more difficult to interpret and such results are best used in conjunction with results of flexibility and voltage breakdown tests. As is well known, the dissipation factor of many materials decreases as thermal aging

* This is not a specific contract responsibility but was added as its significance became apparent.

produces cross linking. On the other hand, thermal aging can produce conducting ions in some materials like PVC which will increase the dissipation factor. In the subject work, the dissipation factor of PVC increased when aged at 120 C in vacuum but did not change significantly with aging in air. It is suggested that the loss of volatile plasticizer removes the stabilizer which would prevent the thermal degradation of polyvinyl chloride. In consequence HCl ions developed in vacuum aging as indicated by the increased dissipation factor. The increase in dissipation factor of Formex with aging in air at 120 C is more difficult to explain. However, these changes correlate with changes in flexibility and voltage breakdown.

The dissipation factor values of all the wire insulation both unaged and aged are very low at cryogenic temperatures - at liquid helium temperature so very low as to make comparisons rather meaningless. It is apparent that these low dielectric losses may prove to be functionally useful.

Moisture Aging

The data on the effect of moisture exposure are not nearly as extensive so far as the data on the effects of thermal aging. However, it would seem that electrical properties at room temperature provide evidence of moisture absorption. Properties at cryogenic temperatures will probably not provide useful indications of absorbed moisture but instead may provide a measure of molecular change caused by hydrolytic degradation. The ability to separate the effects of moisture exposure (a reversible effect with drying) from hydrolytic scission (a permanent degradation) may prove to be very useful.

The limited data indicate effects of moisture absorption in Formex and asbestos insulation and the likelihood of hydrolytic degradation in Formex and ML.

Ribbon Cable

The need for further development of ribbon cable based on H films for cryogenic applications is evident from the results shown to date. If evaluation techniques for films by themselves can be developed, the progress with ribbon cable should be aided.

OBSERVATIONS AND SUMMARY OF TEST RESULTS

Wire Evaluation

Flexibility

Previous studies have shown that decreased flexibility at very low temperatures is the principal limitation in the cryogenic application of insulated wire. Since thermal aging also causes a reduction in flexibility, it is obvious that the effect of thermal aging followed by flexing at cryogenic temperatures should be investigated to determine if the degradation is cumulative.

Results of repeated mandrel flexibility tests in liquid helium are summarized in Table I for several insulated wires as received and after aging in air at 120 and 250 C and in vacuum at 120 C. The determination of failure in flexure is precise with extruded Teflon and PVC since as the mandrel size is decreased, a limit is reached at which cracking and lifting of the insulation suddenly occurs. For the other wires a transition exists between such catastrophic failure and no damage. With Formex, very fine cracking or crazing develops in the film coating when the wire in liquid helium is flexed around a range of mandrel sizes intermediate between a large one which causes no damage and a smaller one which produces crack separation and lifting of the film. With ML, "welts" appear in the film coating on the inside of the bending radius even when bent at room temperature around the smaller mandrels. The film is not always crazed or cracked at the welts. The welts occur only after the wire has been aged at 250°C and appear to result from the loss of adhesion between the film coating and the oxidized copper underneath it. Unless the crazing or welts actually "open up" they usually will not interfere with the functional performance at cryogenic temperatures. At such temperatures, moisture and dirt cannot penetrate as they could at normal temperatures to cause serious degradation of the electrical properties.

When felted asbestos insulated wire is bent around small mandrels at both room and cryogenic temperatures, the felted structure may "tear" to produce cracks. Such cracks are considered to cause functional degradation when they open enough so as to reveal the bare conductor through them.

When Table I is studied it is apparent that thermal aging even at 250 C has no significant effect on the flexibility of extruded Teflon and relatively little effect on felted asbestos coatings. Some degradation of the ML coating occurs only at 250 C and after 120 days exposure G.E. ML seems to be slightly superior to Phelps Dodge ML. As mentioned earlier, the degradation of ML coatings probably results at least in part from the loss of adhesion to the conductor caused by oxidation of the copper which would not occur in many space environments. The Formex coating as expected is charred at 250 C. The comparison of Formex after aging at 120 C in air and vacuum is most interesting. Even after 120 days in vacuum at 120 C, Formex ages very little. Throughout most of the aging period, a vacuum of 2×10^{-6} Torr was held. In air at 120 C aging is apparent after 60 days and probably less. It is apparent that oxidation rather than the loss of volatile components is responsible for the thermal degradation of Formex. Since unaged PVC is so brittle at liquid helium temperature, no additional effect of thermal aging could be observed. However, the color of the PVC coating changes to brown when aged in vacuum but exhibits very little color change when aged in air at 120 C. The significance of such color changes has not been determined with the measurement of flexibility.

Table I

Repeated Mandrel Flexibility Test at 40°

Test Conditions: 10 Reverse Bends Around Mandrel Diameter Shown
While Under Liquid Helium, Failure Indicated
By Low Power Microscope Visual Examination for
Breaks, Cracks, Splits or Separation of Dielectric
From Wire

Insulated Wire	(As Received)	Pre-Condition Aging of Wires Before Repeated Mandrel Flexibility Test in Liquid Helium									
		120 C/Vac 20 Days	120 C/Vac 60 Days	120 C/Vac 120 Days	120 C/Air 60 Days	120 C/Air 120 Days	250 C/Air 60 Days	250 C/Air 120 Days	80 C/95% R.H. 15 Days		
Teflon (Extimided) (0.0114" Wall)	Failure Satisfactory	1" 1-1/4"	1-1/4" 1-1/2"	3/4" 1"	1-1/4" 1-1/2"	3/4" 1"	1" 1-1/4"	3/4" 1"	---	---	---
Poly Vinyl Chloride (0.0071" Wall)	Failure Satisfactory	1-3/4" ---	1-3/4" ---	1-3/4" ---	1-3/4" ---	1-3/4" ---	(No Test) (Above plastic flow temperature)	(No Test) (Above plastic flow temperature)	1-3/4" ---	---	---
Heavy Formex (0.0013" Wall)	Failure Cracked Satisfactory	1/2" 1-1/4" 1-1/4"	1/2" 1-1/2" 1-3/4"	1/2" 3/4" 1-1/4"	1-3/4" ---	1-3/4" ---	(Insulation Charred and Flaked Off at 250 C)	(Insulation Charred and Flaked Off at 250 C)	1-3/4" ---	---	---
HPL (G.E. Co.) (0.0014" Wall)	Failure Welds Satisfactory	None 1/8"	None 1/8"	None 1/8"	None 1/8"	None 1/8"	1/2" 1/8"	1/2" 1/8"	1/4" ---	---	---
HPL (Phelps-Dodge) (0.0011" Wall)	Failure Welds Satisfactory	None 1/8"	None 1/8"	None 1/8"	None 1/8"	None 1/8"	1/2" 1/8"	1/2" 1/8"	1/2" ---	---	---
Asbestos (Phosphate) (0.0054" Wall)	Failure Cracks Satisfactory	1/2" 1/8" 3/4"	1/4" 1/2" 3/4"	3/4" 1/2" 1"	1/4" 3/4"	1/4" 3/4"	1/2" 1/8"	1/2" 1/8"	3/4" 1"	---	---
Asbestos (VL Overcoat) (0.0064" Wall)	Failure Cracks Satisfactory	1/4" 1/8" 1/2"	1/2" 1/8" 1/2"	1/2" 1/8" 1/2"	1/4" 3/4"	1/4" 3/4"	1/2" 1/8"	1/2" 1/8"	3/4" 1"	---	---
Triple ML (Phelps-Dodge) (0.0016" Wall)	Failure Satisfactory	None 1/8"	None 1/8"	None 1/8"	None 1/8"	None 1/8"	1/2" 1/8"	1/2" 1/8"	1/4" ---	---	---

The effect of aging 15 days at 80 C and 95% RH before flexure tests in liquid helium is shown in the column of Table I. A definite degradation in flexibility after moisture exposure is apparent with ML and particularly with Formex. Great care was taken to prevent moisture condensation on the outside of the samples before they were immersed in liquid helium so as to avoid the formation of ice. It is believed that the degradation observed results from hydrolytic degradation of the molecular structure to which both Formex and ML are known to be susceptible. However, the limited amount of molecular degradation occurring in 15 days at 80 C and 95% RH would be difficult to detect with conventional means. If, in fact, hydrolytic degradation is involved, cryogenic flexibility is a very sensitive test for it.

Table II describes results of flexibility measurements on unaged and aged wires made at room temperature. In one approach the wires were wrapped on their own diameter - 1X. In the other approach the wires were subjected to repeated reverse flexibility tests about mandrels of different sizes using the same equipment as used for tests in liquid helium. The flexibility tests at room temperature do not detect any effect of thermal or moisture aging on either Teflon or PVC extruded coatings. However, the effect of thermal aging at 250 C on ML is evident. The decrease in room temperature flexibility of Formex aged in air at 120 C is evident but is not evident when the aging takes place in vacuum (confirming the measurements made at liquid helium temperature - Table I.) There is an indication that flexibility at room temperature of asbestos insulated wire degrades slightly after aging at 250 C.

The moisture aging shown in the last column appears to have no significant effect on the flexibility of any of the wires. The degradation indicated with Formex and ML at cryogenic temperatures is not obvious at room temperature.

In further investigation, wires were wound about their own diameter - 1X, or back again - 3X, or even back again the second time - 5X. The wires so wound were then immersed in liquid helium to see if film failure might occur in this strained condition which did not develop at room temperature as originally flexed. In only one case - a 5X bend for HF aged 60 days in air at 120 C - was damage apparent after immersion in liquid helium that was not noticed before immersion.

Throughout the work, it has been apparent that mandrel flexibility measurements may be useful in determining the cure of ML enamel which is so difficult to measure in more conventional ways. In Table IV, mandrel flexibility measurements at liquid helium temperature are shown for ML wire cured with two different oven temperatures at three oven speeds. A sharp demarcation for different curing conditions occurs as indicated by the considerable change in flexibility at liquid helium temperature. These differences in cure are not indicated by flexibility tests made at room temperature.

Table II

Repeated Mandrel Flexibility Test at 25°C

(Test Conditions: 10 Reverse Bends Around Mandrel Diameter Shown)

<u>Insulated Wire</u>	<u>Overall Wire Dia. - In.</u>	<u>Pre-Conditioning Before Test</u>			
		<u>120 C/Vac 120 Days</u>	<u>120 C/Air 120 Days</u>	<u>250 C/Air 120 Days</u>	<u>80 C/95% R.H. Air 15 Days</u>
Teflon (Extruded) (0.0114" wall)	Failure Satisfactory	OK - 1X* 1/4"	OK - 1X 1/2"	OK - 1X 1/2"	OK - 1X (No test)
Polyvinyl Chloride (0.0071" wall)	Failure Satisfactory	OK - 1X 1/8"	OK - 1X 1/8"	OK - 1X (No test)	OK - 1X (No test)
Heavy Formex (0.0013" wall)	Failure Satisfactory	OK - 1X 1/8"	1/4" 3/4"	(No test) (No test)	OK - 1X 1/8"
HML (G.E. Co.) (0.0014" wall)	Failure Welts Satisfactory	OK - 1X -- 1/8"	OK - 1X -- 1/8"	1/4" 1/2" - 3/4" 1"	OK - 1X -- 1/8"
HML (Phelps-Dodge) (0.0011" wall)	Failure Welts Satisfactory	OK - 1X -- 1/8"	OK - 1X -- 1/8"	1/2" 3/4" - 1" --	OK - 1X -- 1/8"
Asbestos (Phosphate) (0.0054" wall)	Failure Cracks Satisfactory	3/4" 1/8" - 3/4" 1"	3/4" 1/8" - 3/4" 1"	1" 1/8" - 1" --	1" 3/4" - 1" --
Asbestos (ML over- coated) (0.0064" wall)	Failure Cracks Satisfactory	1/8" 1/8" - 3/4" 1"	1/8" 1/4" - 1/2" 3/4"	1/4" 1/8" - 1/4" 1/2"	1/4" 1/8" - 1/2" 3/4"

* OK - 1X, the wire can be wrapped on its own diameter without visible damage.

Table III

Effect of Pre-Conditioned Insulated Wires on the
Flexibility Test at 25C/4K

Test Condition: Wire Wound on Itself at Room Temperature
and then Immersed in Liquid Helium for
15 Minutes Before Visual Examination for
Breaks, Cracks, Splits or Separation of
the Dielectric from the Wire

<u>Wire and Pre-Conditioning</u>	<u>Flexibility at 25 C + Immersion in Liquid Helium</u>
<u>Teflon (Extruded, 0.0114" Wall)</u>	
Original	OK - 1X*
20 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Vacuum	OK - 1X
120 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Air	OK - 1X
120 Days, 120 C, Air	OK - 1X
60 Days, 250 C, Air	OK - 1X
120 Days, 250 C, Air	OK - 1X
<u>Polyvinyl Chloride (0.0071" Wall)</u>	
Original	OK - 1X
20 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Vacuum	OK - 1X (After 60 days in vacuum at 120°C, wire turned brown in color.)
120 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Air	OK - 1X
120 Days, 120 C, Air	OK - 1X
60 Days, 250 C, Air	(Not run)
120 Days, 250 C, Air	(Not run)
<u>Heavy Formex (0.0013" Wall)</u>	
Original	OK - 1X
20 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Vacuum	OK - 1X
120 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Air	Failed - 3X** at room temp. -(1 crack at 5X in liquid He)
120 Days, 120 C, Air	Failed - 5X at room temp.
60 Days, 250 C, Air	(Formex charred - no test)
120 Days, 250 C, Air	(Formex charred - no test)

* OK - 1X, the wire can be wrapped on its own diameter and subsequently immersed in
liquid He without visible damage.

** 3X, the wire is wrapped on itself and then back again on itself.

Table III (Contd.)

Effect of Pre-Conditioned Insulated Wires on the
Flexibility Test at 25C/4K

<u>Wire and Pre-Conditioning</u>	<u>Flexibility at 25 C + Immersion in Liquid Helium</u>
<u>HML (G.E. Co., 0.0014" Wall)</u>	
Original	OK - 1X
20 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Vacuum	OK - 1X
120 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Air	OK - 1X
120 Days, 120 C, Air	OK - 1X
60 Days, 250 C, Air	Inside Radial Welts (Developed at 23 C)
120 Days, 250 C, Air	Inside Radial Welts (Developed at 23 C)
<u>HML (Phelps-Dodge, 0.0011" Wall)</u>	
Original	OK - 1X
20 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Vacuum	OK - 1X
120 Days, 120 C, Vacuum	OK - 1X
60 Days, 120 C, Air	OK - 1X
120 Days, 120 C, Air	OK - 1X
60 Days, 250 C, Air	Inside Radial Welts (Developed at 23 C)
120 Days, 250 C, Air	Inside Radial Welts (Developed at 23 C)
<u>Asbestos (Phosphate, 0.0054" Wall)</u>	
Original	Failed - 1X (During Winding at 23 C)
20 Days, 120 C, Vacuum	Failed - 1X (During Winding at 23 C)
60 Days, 120 C, Vacuum	Failed - 1X (During Winding at 23 C)
120 Days, 120 C, Vacuum	Failed - 1X (During Winding at 23 C)
60 Days, 120 C, Air	Failed - 1X (During Winding at 23 C)
120 Days, 120 C, Air	Failed - 1X (During Winding at 23 C)
60 Days, 250 C, Air	Failed - 1X (During Winding at 23 C)
120 Days, 250 C, Air	Failed - 1X (During Winding at 23 C)
<u>Asbestos (ML Coated, 0.0064" Wall)</u>	
Original	Failed - 1X (During Winding at 23 C)
20 Days, 120 C, Vacuum	Failed - 1X (During Winding at 23 C)
60 Days, 120 C, Vacuum	Failed - 1X (During Winding at 23 C)
120 Days, 120 C, Vacuum	Failed - 1X (During Winding at 23 C)
60 Days, 120 C, Air	Failed - 1X (During Winding at 23 C)
120 Days, 120 C, Air	Failed - 1X (During Winding at 23 C)
60 Days, 250 C, Air	Failed - 1X (During Winding at 23 C)
120 Days, 250 C, Air	Failed - 1X (During Winding at 23 C)

Table IV

Effect of Curing Conditions of ML Coated Wire on Flexibility

Flexibility Test Conditions: 10 Reverse Bends Around Mandrel in Liquid Helium, Failure Indicated by Breaks, Cracks or Separations of the Dielectric by Visual Observations with a Low Powered Microscope

<u>Oven Temperature</u>	<u>Wire Speed</u>	<u>Flexibility</u>		<u>Flexibility (Wire Wound on Itself at Room Temperature and then Immersed in Liquid Helium for 15 Minutes)</u>
		<u>Failure</u>	<u>Satisfactory</u>	
High	Slow	None	1/8"	OK - 1X
Moderate	Intermediate	None	1/8"	OK - 1X
High	Intermediate	None	1/8"	OK - 1X
High	Intermediate	None	1/8"	OK - 1X
High	Fast	1/4"	1/2"	OK - 1X
Moderate	Fast	1/4"	1/2"	OK - 1X
High	Fast	1/4"	1/2"	OK - 1X

Compression Cut Through

As described in the sixth quarterly report dated January 15, 1963, PVC extruded coatings failed in compression in liquid nitrogen (under the influence of a 4.7 load applied to a .025 in. diameter rod perpendicular to the wire). In a similar test Formex insulation cracked and spalled as a 50-pound load was applied, but no means of determining the point of failure was developed.

ML insulation was not damaged even when 70 pounds was applied to a .025 in. diameter rod. Under such conditions the ML coated wire was deformed considerably as shown in Photo 1.

Principal attention has been directed to the development of a means for detecting failure when it occurs in crushing or cut-through tests. Up to 700 volts was applied between the Formex conductor and the steel rod in the hope that voltage breakdown would indicate when fracture of the Formex film occurred as load was applied to the mandrel while immersed in liquid nitrogen. Even though considerable cracking and spalling was obtained, no voltage failure resulted. Apparently the liquid nitrogen impregnated the cracked insulation which provided sufficient spacing so that dielectric failure could not be obtained even at 700 volts (higher voltages could not be applied easily because of equipment limitations).

In consequence, attempts were made to develop a crushing cut-through test which would disrupt the insulation sufficiently so that actual electrical contact would be made between mandrel and insulated conductor. It was expected that more severe tests might also cause failure of ML insulation. None of the tests developed accomplished the desired objectives but are described in the following since the details give some idea of the parameters of the problem.

The crossed mandrel test (see Fig. A) was repeated using three mandrel diameters - .010", .025" and 0.250". Stress-strain plots of the compression were plotted with an Instron Tester. The failure of the relatively thick extruded PVC could be detected but no reliable indication of failure in relatively thin coatings like Formex seemed possible. In the tests sketched in Figs. B and C, the mandrel was replaced with a hard steel ball - 0.12 or .030 in. in diameter. In both tests the copper deformed. Formex shattered without external electrical or mechanical indication of failure. ML coatings did not fail. In the test shown in Fig. C the small indenting ball tended to slip between the two insulated wires which were clamped together in parallel. It was hoped that this shearing action would produce metal conduct, but it did not.

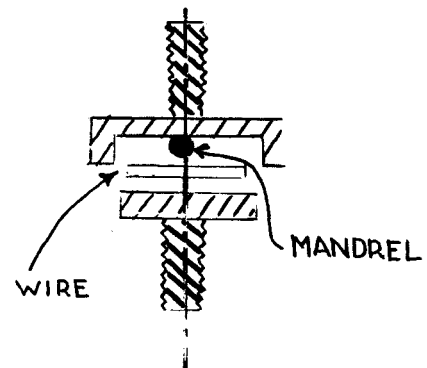


FIG. A

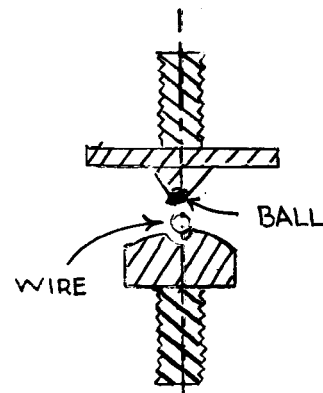


FIG. B

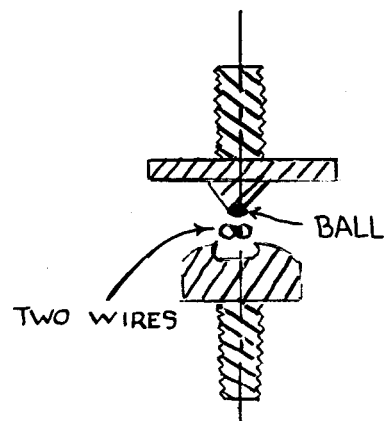


FIG. C

In the arrangement shown in Figs. D and E, two insulated wires were each shaped into a U configuration, crossed and subjected to compression. An over-and-under arrangement was used also as illustrated in Fig. E. The copper deformed at the four points of contact shattering Formex in some cases but never ML. Here again it was impossible by either electrical or mechanical means to determine the load at which failure occurred. The U configuration was perhaps easier to control than the mandrel or penetrating ball test.

In a final attempt, four wires were held in grooves on the surface of a wedge arranged shown in cross section in Fig. F. A wedge angle of 3° was used since larger angles produced uncontrolled slippage. Even at a 3° angle the test is difficult to control. Unfortunately, the hoped-for electrical contact between the failed samples after increments of increasing load did not occur.

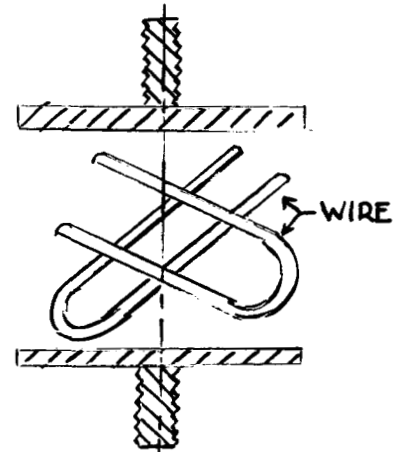


FIG. D

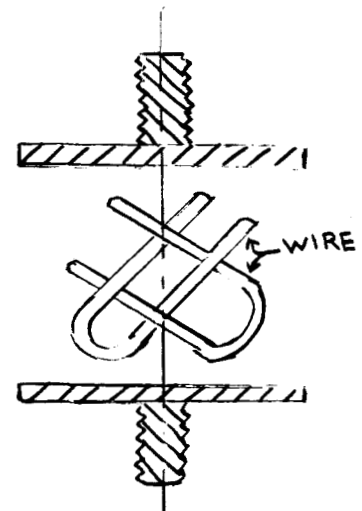


FIG. E

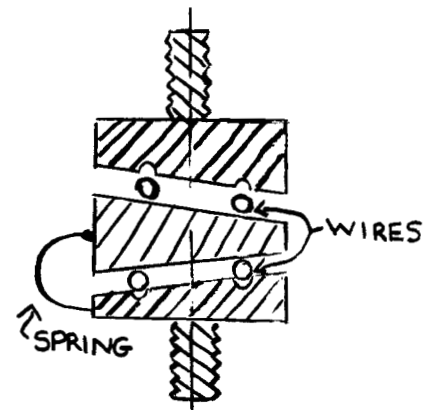


FIG. F

Voltage Breakdown - Effect of Thermal Aging

Voltage breakdown, like flexibility, can be used as a tool to measure the degree of thermal and moisture degradation in insulated wire. The effect of thermal and moisture aging on the voltage breakdown of twisted pairs both at room and at 4.2°K is shown in Table V. The three columns at the right of the table summarize the effects of moisture and will be discussed later. The remainder of the table is devoted to the effects of thermal aging at 175 C in both vacuum and air and at 250 C in air.

Voltage breakdown values are characteristically quite variable so that the range of the values (min. - max.) as well as the average value is tabulated. With such variable results, it is difficult to grasp their significance in tabulated form. Consequently, the data are plotted (as solid lines for breakdown tests made at 23 C and as dotted lines for tests made in liquid helium, 4.2°K) in several figures as follows:

- Fig. 1 - Heavy Formex Wire (HF) aged in vacuum and air at 120 C.
- Fig. 2 - G.E. heavy ML wire aged in vacuum and air at 120 C.
- Fig. 3 - Phelps Dodge heavy ML Wire aged in vacuum and air at 120 C.
- Fig. 4 - G.E. and PD ML wires aged in air at 250 C.
- Fig. 5 - Extruded Teflon insulated wire aged in vacuum and air at 120 C.
- Fig. 6 - Extruded Teflon insulated wire aged in air at 250 C.
- Fig. 7 - Extruded polyvinylchloride insulated wire aged in vacuum and air at 120 C.

These figures will be discussed individually.

From Fig. 1 it is apparent that for Formex no significant difference exists between breakdown measurements made at room (solid lines) and at 4.2 K (dotted lines). No effect of thermal aging at 120 C in vacuum is obvious but in air a slight but barely significant drop in breakdown voltage is evident. The effect of aging in air at 120 C on the breakdown voltage of Formex is not nearly as obvious as the decrease in flexibility described earlier in this report.

In Figs. 2 and 3 it can be seen that for ML (unlike Formex) the breakdown voltage at 4.2 K is significantly lower than at room temperature. No reason for this difference is easily postulated. However, no effect of thermal aging at 120 C on ML is significant in either air or vacuum. In fact, even when aged at 250 C in air (Fig. 4), no significant effect of thermal aging is apparent. The breakdown voltage of the Phelps Dodge ML is lower than the P.D. wire but it should be noted from Table V that the nominal insulation thickness of the G.E. wire is .0014 in. and

Table V

Summary of Thermal and Humidity Aging Effects on the Voltage Breakdown (KV)
Of Insulated Wires at Room and Cryogenic Temperatures

Insulated Wire (Temperature of Test) →	Original 25C AK	Thermal Aging												Moisture Aging for 15 Days (95% Relative Humidity at 80 C)					
		Vacuum at 120 C				Air at 120 C				Air at 250 C				Tested at:					
		25C	AK	25C	AK	60	25C	AK	120	25C	AK	60	25C	AK	120	25C	AK	25C	AK
Heavy Formex (0.0013" Wall)	Avg. Min. Max.	7.1 6.1 8.8	8.0 7.9 8.1	8.5 8.4 8.6	7.6 6.7 8.3	7.8 7.8 7.8	7.8 6.8 8.9	7.1 6.2 8.4	7.5 6.8 8.2	5.4 4.6 6.8	5.6 4.6 6.7	5.7 4.9 6.7	4.5 3.4 5.3	(Charred off at 250 C)				3.3 3.1 3.4	5.9 4.2 7.6
Heavy ML (G.E.) (0.0014" Wall)	Avg. Min. Max.	13.3 11.4 14.6	9.0 8.7 9.4	16.6 15.6 17.4	9.8 9.7 9.8	15.9 14.0 17.8	8.4 8.4 9.0	15.6 11.6 17.6	8.9 8.8 9.0	14.2 12.5 15.6	10.1 9.4 10.6	13.1 10.2 15.6	7.6 7.6 15.6	14.1 10.4 17.2	8.5 8.3 8.6	13.6 11.6 14.6	8.3 8.1 8.6	14.3 14.0 14.6	7.3 5.0 9.5
Heavy ML (Phelps-Dodge) (0.0011" Wall)	Avg. Min. Max.	10.5 9.1 11.7	7.2 5.0 9.6	10.9 9.3 12.2	8.7 8.3 9.0	11.5 11.3 11.6	6.8 4.7 7.8	11.9 10.2 13.2	5.8 4.4 7.2	10.1 8.0 12.1	7.4 7.3 7.5	10.4 9.5 12.0	7.2 6.8 8.8	8.7 8.1 9.3	7.4 6.8 7.8	10.0 9.7 10.3	7.6 7.6 7.7	9.0 7.4 10.2	8.0 8.0 4.3
Teflon (Extruded) (0.0114" Wall)	Avg. Min. Max.	18.9 15.8 20.4	17.4 16.2 19.7	18.7 17.4 19.4	17.7 15.8 19.8	17.1 15.6 18.8	18.5 14.6 20.5	18.3 15.6 20.4	19.5 18.6 20.5	15.8 12.6 19.2	19.6 18.8 20.5	17.4 17.0 18.2	19.3 20.5 19.4	13.1 12.8 13.4	17.2 16.2 18.5	13.1 11.6 15.4	17.0 13.4 19.4	19.9 19.4 20.3	17.4 17.4 20.3
Poly Vinyl Chloride (0.0071" Wall)	Avg. Min. Max.	>20.5 10.2 14.6	>20.5 12.9 10.2	>20.5 10.2 14.6	>20.5 10.6 5.0	>20.5 10.6 13.8	6.5 4.2 8.2	>20.5 20.5 13.8	6.5 4.2 8.2	>20.5 16.9 16.2	16.9 16.2 17.6	>20.5 17.7 17.4	17.7 17.4 18.4	(Not aged at 250 C)				18.0 16.6 20.5	16.6 16.6 20.5
Asbestos (Phosphate) (0.0054" Wall)	Avg. Min. Max.	1.4 1.4 1.5	2.5 2.4 2.6	1.0 2.0 2.3	1.0 0.7 2.3	1.0 1.7 1.1	1.8 1.6 1.9	1.0 0.9 1.1	1.8 1.6 1.9	1.1 1.1 2.0	1.8 1.6 2.0	1.0 1.0 1.9	1.8 1.7 2.2	1.0 0.9 1.1	1.9 1.7 2.0	1.0 0.9 2.0	2.1 2.0 2.2	1.0 1.0 2.3	2.3 2.2 2.3
Asbestos (ML Coated) (0.0064" Wall)	Avg. Min. Max.	1.3 1.2 1.4	2.5 2.4 2.6	1.1 1.0 1.2	2.4 2.3 2.4	1.2 1.1 1.3	2.6 2.3 2.5	1.1 1.1 1.3	2.6 2.5 2.6	1.1 1.0 1.2	2.3 2.2 2.6	1.1 1.0 1.1	2.4 2.3 2.6	1.2 1.1 1.3	2.4 2.3 2.6	1.1 1.0 1.2	2.4 2.3 2.4	1.4 1.4 2.4	0.5 0.5 0.5
Triple ML (Phelps-Dodge) (0.0016" Wall)	Avg. Min. Max.	13.6 11.2 15.1	11.2 10.8 11.5	13.6 11.2 15.1	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5	11.2 10.8 11.5

Test Conditions: Average of 3 Breakdowns (20 KV/Minute Rise, 60 Cycles) of
Twisted Pairs of Wire in Room Temperature Air or Under
Liquid Helium at 4K.

the PD wire only .0011 which can easily account for the difference in the absolute values of the breakdown voltage. The effect of thermal aging on the General Electric and Phelps Dodge wires is similar.

From Fig. 5 it is apparent that, like Formex, the breakdown of extruded Teflon is similar at room temperature and at 4.2°K. As expected, there seems to be no effect of thermal aging at 120 C in either air or vacuum. The results in Fig. 6 are puzzling. It would seem that the room temperature breakdown of Teflon is slightly degraded with aging at 250 C while the breakdown values obtained at 4.2 K in liquid helium exhibit no significant change with such aging.

The effect of thermal aging on the voltage breakdown of polyvinylchloride insulated wire as shown in Fig. 7 is startling. Breakdown measurements made at room temperature all remain above 20.5 Kv (the upper limit of voltage available) despite aging at 120 C in either vacuum or air. However, breakdown measurements made at 4.2 K in liquid helium indicate a marked and increasing effect of thermal aging at 120 C in vacuum and a small but significant decrease with aging in air. It should be noted that when aged in vacuum the PVC showed a pronounced color change from yellow to brown, but when aged in air, only a very slight darkening of the yellow color. It is likely that plasticizer volatilized more rapidly under vacuum. The effect of plasticizer loss did not produce measurable increased stiffness at 23 C as shown earlier. In liquid helium even unaged PVC is too brittle to be evaluated. Aged PVC, when flexed at room temperature and then immersed in liquid helium, gave no visible indication of damage. (See Table III) It is postulated that the homogeneity of the PVC aged in vacuum is, in fact, damaged by immersion in liquid helium as indicated by the degradation in electrical breakdown even though the effect of aging is not visible to the eye except as a color change.

Voltage Breakdown - Effect of Moisture Aging

In order to determine the effect of moisture, the contract requires exposure at 80°C and 95% RH for 15 days - a very severe and accelerated condition which may not only result in physical absorption of water but also in chemical degradation (hydrolytic scission) of some materials. The contract requires breakdown measurements only at room conditions after moisture exposure and these are reported in Table V. In addition, the breakdown values in liquid helium after moisture exposure are reported also. The humidity cabinet was cooled carefully to room temperature before opening so as to avoid condensation and the samples tested as rapidly as possible after removal. Unavoidably, a few minutes elapsed between removal of the samples from the humidity chamber and the time of voltage breakdown. From Table V it can be seen that the moisture exposure decreased the room temperature breakdown voltage of Formex, PVC, and the phosphate impregnated asbestos. It is probable that the ML coated asbestos insulation dried out very rapidly so that any effect of absorbed moisture could not be determined. Apparently moisture exposure did not adversely affect ML or extruded Teflon. When tested at 4.2 K the Formex exposed to moisture exhibits a higher voltage breakdown than when tested

at room temperature but not as high as for samples not exposed to moisture. Since absorbed moisture would not be expected on the basis of other evidence to decrease the breakdown voltage at liquid helium temperature, it is expected that the decrease shown results from molecular degradation caused by hydrolytic scission. Such hydrolytic scission was believed responsible for degradation in the cryogenic flexibility of both Formex and ML as described earlier. However, hydrolytic degradation of ML is not indicated by the breakdown voltage values.

It is recognized that the breakdown measurements should be made in the humidity chamber and not after removal when drying can occur. Measurements of this kind were intended but trouble with lead breakdown occurred. A few measurements were made successfully with Phelps Dodge ML and inorganic bonded asbestos as shown in Table V. In this case, the breakdown voltage was decreased due to the influence of absorbed moisture. A new humidity chamber and lead arrangement have been developed to overcome these problems as shown in Sketch #1 and a new series of tests is underway.

Capacitance and Dissipation Factor

The measurement of capacitance and dissipation factor after various aging periods at several temperatures described in the sixth quarterly report has been continued in this report. The dissipation factor data of Table IV of the sixth quarterly report, together with additional data, have been plotted in this report as Table VI. Several observations on these rather extensive data can be made. All of the values of dissipation factor measured with samples in liquid helium at 4.2 K are very low - so low, in fact, as usually to be below the range of a very sensitive bridge and where values are reported there is reason, in some cases, to believe they may be in error (such measurements are difficult to make). Consequently, the measurements at 4.2 K provide little basis for comparison. While the values for samples measured in liquid nitrogen are still very low, it does seem possible to make comparisons at this temperature. However, the best comparison can apparently be made between the room temperature values before and after heat aging.

All of the dissipation factor values for Teflon are very low, but in general, the values at room temperatures decrease with thermal aging. It is suggested that thermoplastic flow has occurred even at 120 C so that the cabled sample has loosened somewhat. Such changes may, in fact, be responsible for some of the questionable results. With PVC, very definite and interesting trends can be observed. The dissipation factor at 23 C decreases with aging at 120 C in air and increases with aging at 120 C in vacuum. These results correlate with the flexibility and voltage breakdown data which indicate degradation of these properties in vacuum but not in air. In the opposite fashion, the dissipation factor values for Formex increase when aged in air at 120 C but show little change with aging in vacuum at the same temperature. Again these results correlate with data which show that aging in air but not in vacuum degrades the flexibility and breakdown voltage of Formex.

Table VI (Contd.)

Effect of Thermal Aging on Dissipation Factor at 100°C

[illegible]

With ML, perhaps the most interesting comparison lies between relatively high values of $\tan \delta$ for unaged G.E. wire as compared to somewhat higher values for the Phelps Dodge product. It is suspected from these data that the G.E. wire has more cure as received which may account for some of the comparisons observed in other properties also. With aging at 120 C in both air and vacuum, the dissipation factor of both ML wires decreases, perhaps indicating still further cure although the temperature seems too low for such effects. In contrast, with both ML wires, aging at 250 C produces an increase in the dissipation factor - perhaps losses caused by the copper oxide at the interface between film coating and conductor are being measured. The changes in dissipation factor with aging of the asbestos insulated wires are believed to be due primarily to loss of moisture; but at 250 C, the development of copper oxide may be a factor in this case also.

In Table V of the sixth quarterly report, values of capacitance after thermal aging have been reported. These values depend so much on sample geometry that they were not considered significant. Examination of the additional capacitance data has not changed this observation so the values are not reported here although they are available.

Additional Breakdown Studies

As required in the contract, breakdown measurements will be made in vacuum at high temperatures. After some study, a relatively simple method for making such measurements has been developed. The apparatus is shown in Sketch #2. Only one sample is tested at a time in an alundum tube which can be heated quickly and a very good vacuum pulled rapidly. Measurements are underway and results will be reported in the next quarterly report.

A special apparatus has also been developed to make voltage breakdown measurements at -60°C as required in the contract. These measurements also should be completed during the next quarter.

Ribbon Cable

In Table VII repeated mandrel flexibility tests 2 in liquid helium are reported for several constructions of ribbon cable. It should be recognized that flexibility depends on adhesion and sample thickness as well as the nature of the materials. Sample E, which is .012 in. thick, does not fail when flexed around a $\frac{1}{4}$ " mandrel. This sample consists of FEP Teflon film on one side and H film on the other, which has been coated with a thin FEP Teflon film to provide the bond under pressure and temperature. It is hoped that a sample with H film on both sides similarly bonded can be obtained. The thinner (.0086) resin bonded H film sample B also performs well in liquid helium. However, the performance of FEP Teflon alone (Sample D) is disappointing.

Recognizing that it would be desirable to study the flexure characteristics of the plastic films by themselves, measurements were made in liquid nitrogen. Surprisingly, in liquid nitrogen it is possible

Table VII

Repeated Mandrel Flexibility Tests of Ribbon Cable

Test Condition: Strips 3/16" Wide Were Carefully Cut from the Cable for Mandrel Winding in a Manner Similar to the Procedure Developed for Round Wire. 10 Reverse Turns in Liquid Helium was the Criterion for Failure

<u>Sample No.</u>	<u>Material</u>	<u>Results</u>	<u>Mandrel Dia. - In.</u>	<u>Remarks</u>
A	Methode Plyoduct (PD-812-P4) (31/32" Wide x 0.012" Thick, 12 Copper Conductors) (Mylar)	Failed Satisfactory	1/2" 1"	(If stress relieved by pre- flexing at Room Temperature)
B	Polystrip (H-100-C-25) (2-5/8" Wide x .0086" Thick, 25 Conductors) (Resin Bonded H Film)	Failed Satisfactory	-- 1/4"	(Could not wind on 1/8" Mandrel) (Except at point of high stress near mandrel attachment)
C	Polystrip (P-100-C-12) (1-5/16" Wide x .0085" Thick, 12 Conductors) (Resin Bonded Mylar)	Failed Satisfactory	1/4" 1/2"	(Except at point of high stress)
D	Polystrip (TX-156-C-20) (3 1/2" Wide x .012" Thick, 20 Conductors) (FEP Teflon)	Failed Satisfactory	1" --	(Test strip 1-1/16" wide)
E	IRC-HX-100-C-12 (1-5/16" Wide x .012" Thick, 12 Conductors) (FEP Teflon/ H Film Bonded with FEP Teflon)	Failed Satisfactory	--	(Could not wind on 1/8" Mandrel)

to crease .010 in. films of FEP, Teflon and Mylar without failure. Consequently, other means of evaluation have been sought. L. Hogue developed the ingenious idea of using a punch in the center of a ring anvil to compare the strain capabilities of films at cryogenic temperatures. Preliminary tests are interesting and if time permits, this concept will be further developed.

Dielectric Properties of Cryogenic Liquids

As mentioned in the monthly report for January 1963, the vacuum capacitor to be used for the dielectric measurements of cryogenic liquids cracked as the glass filling tube was sealed to it. Attempts to repair it proved futile. Consequently, a new vacuum capacitor has been obtained with the glass tube used by the manufacturer to obtain the vacuum left in place. This new capacitor with a nominal value of 1000 picofarads should permit very accurate measurement of capacitance and dissipation factor. It has been provided with guard electrodes, installed in a cryostat and the electrical characteristics checked. Test results should be obtained very shortly.

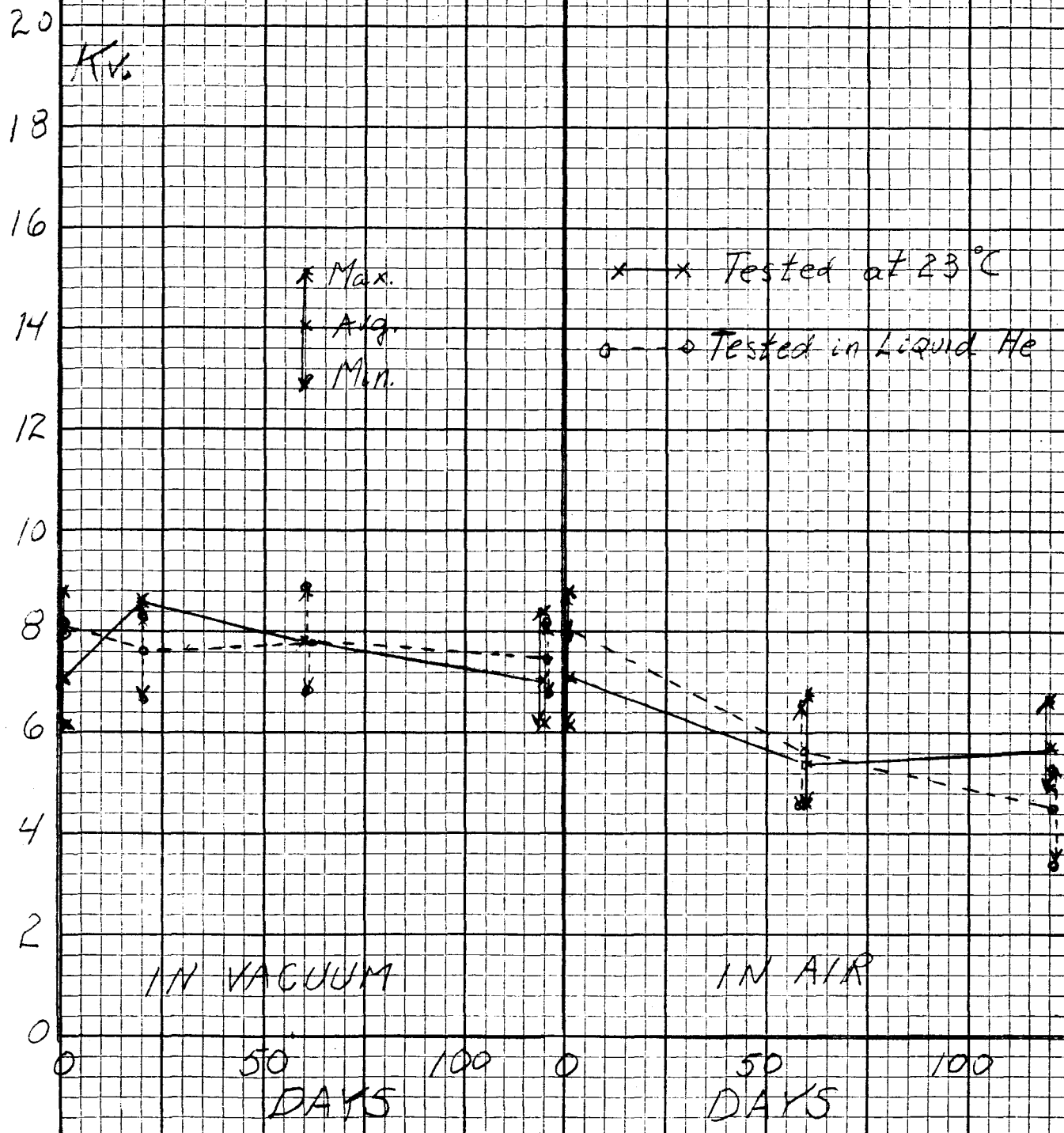
PROGRAM FOR APRIL AND THE EIGHTH QUARTER

In April the principal effort will involve measurements of the electrical properties of cryogenic liquids, the vacuum breakdown of wire samples at high temperatures, the breakdown measurements at -60°C and the measurement of breakdown voltage, resistance and AC properties of wire samples exposed at high humidity.

Unfinished portions of the April program, as well as other contract requirements such as abrasion resistance, will be complete during the coming quarter. It seems reasonable to expect that essentially all of the requirements specified in the contract will be completed.

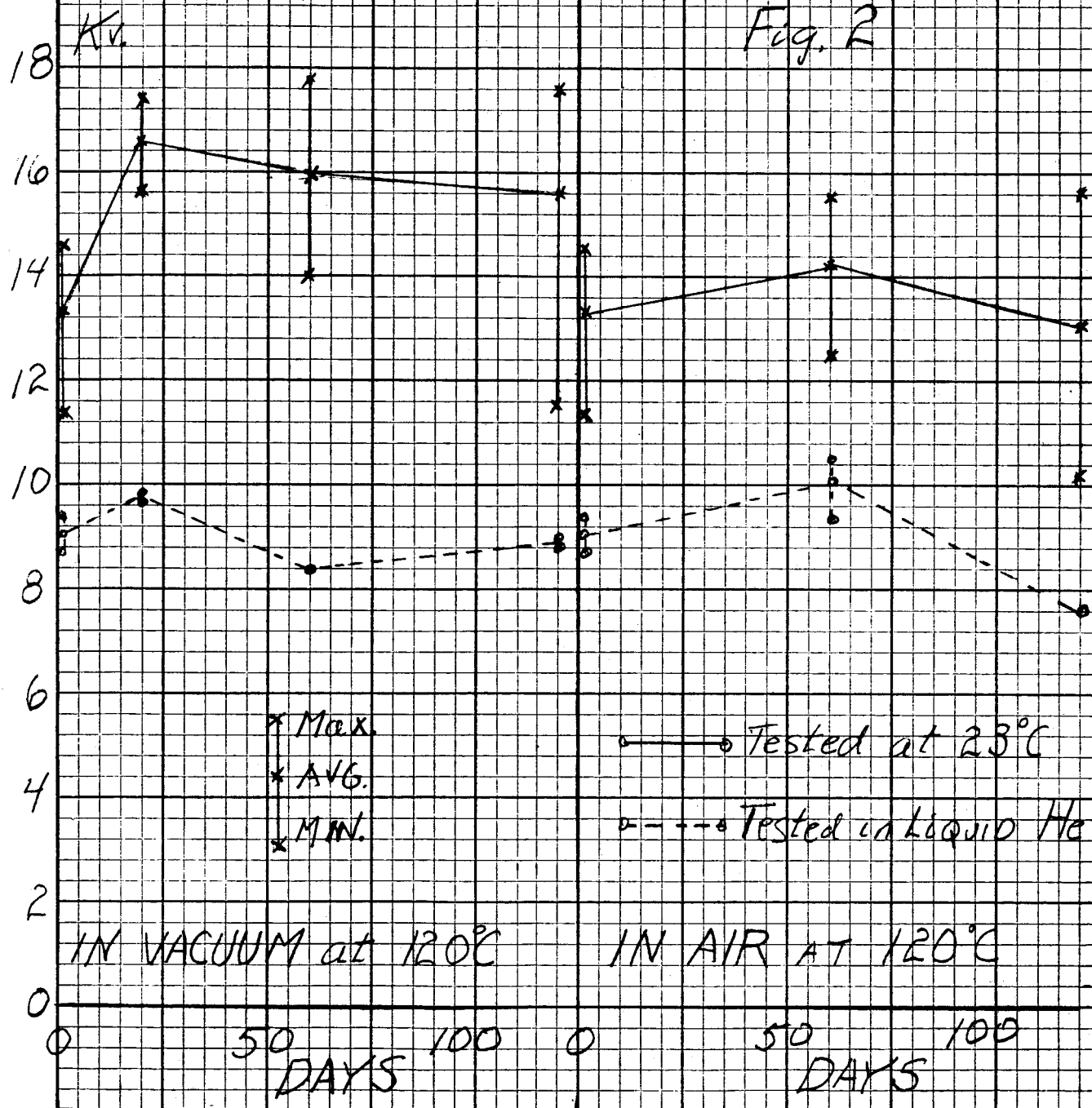
VOLTAGE BREAKDOWN VS. THERMAL AGING AT 120°C HEAVY FORMEX

Fig. 1



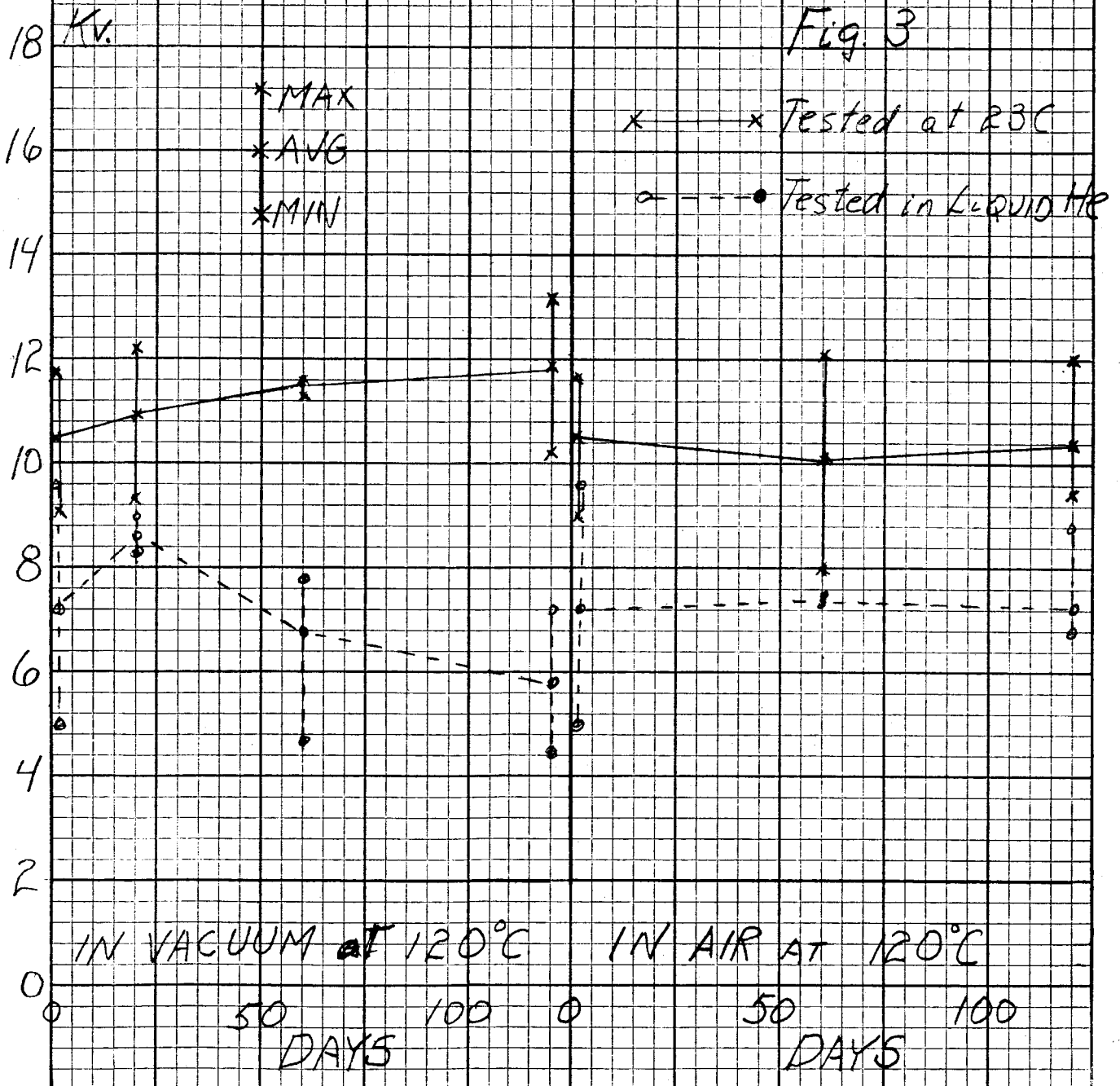
VOLTAGE BREAKDOWN VS THERMAL AGING AT 120°C HEAVY ML (G.E.)

Fig. 2



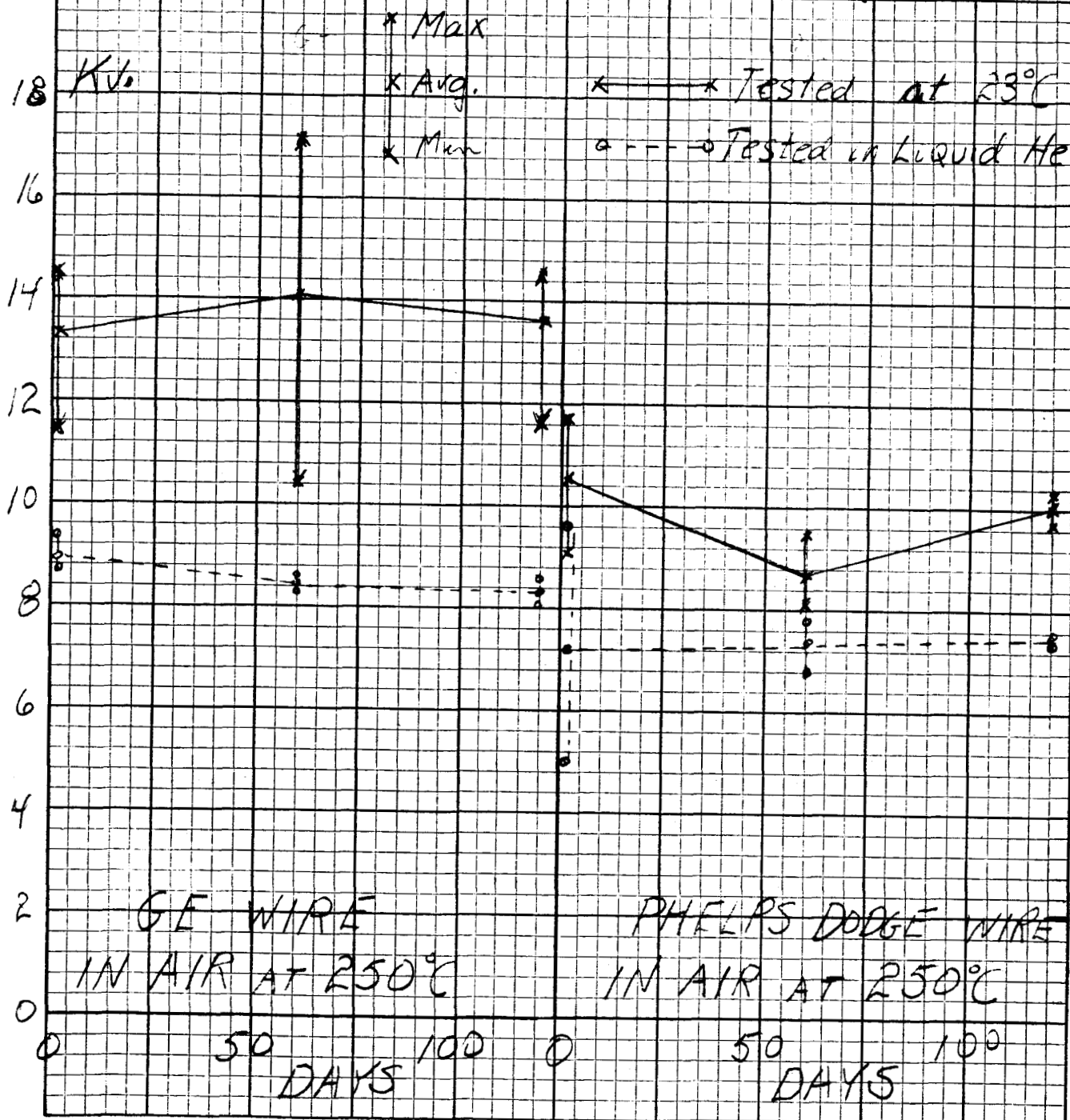
VOLTAGE BREAKDOWN VS. THERMAL AGING AT 120°C HEAVY ML (PHELPS DODGE)

Fig. 3

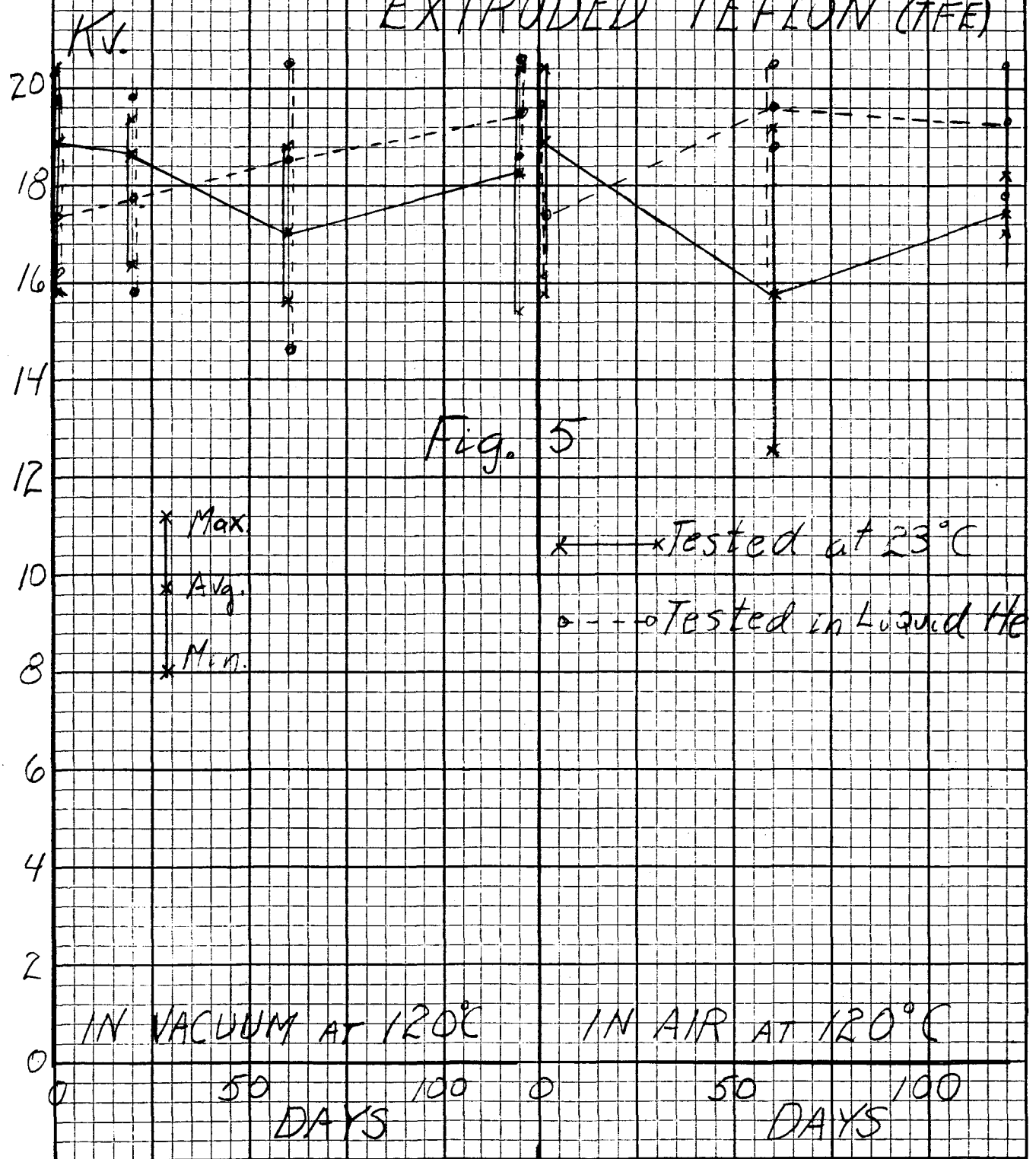


VOLTAGE BREAKDOWN VS. THERMAL AGING AT 250°C HEAVY ML

Fig. 4

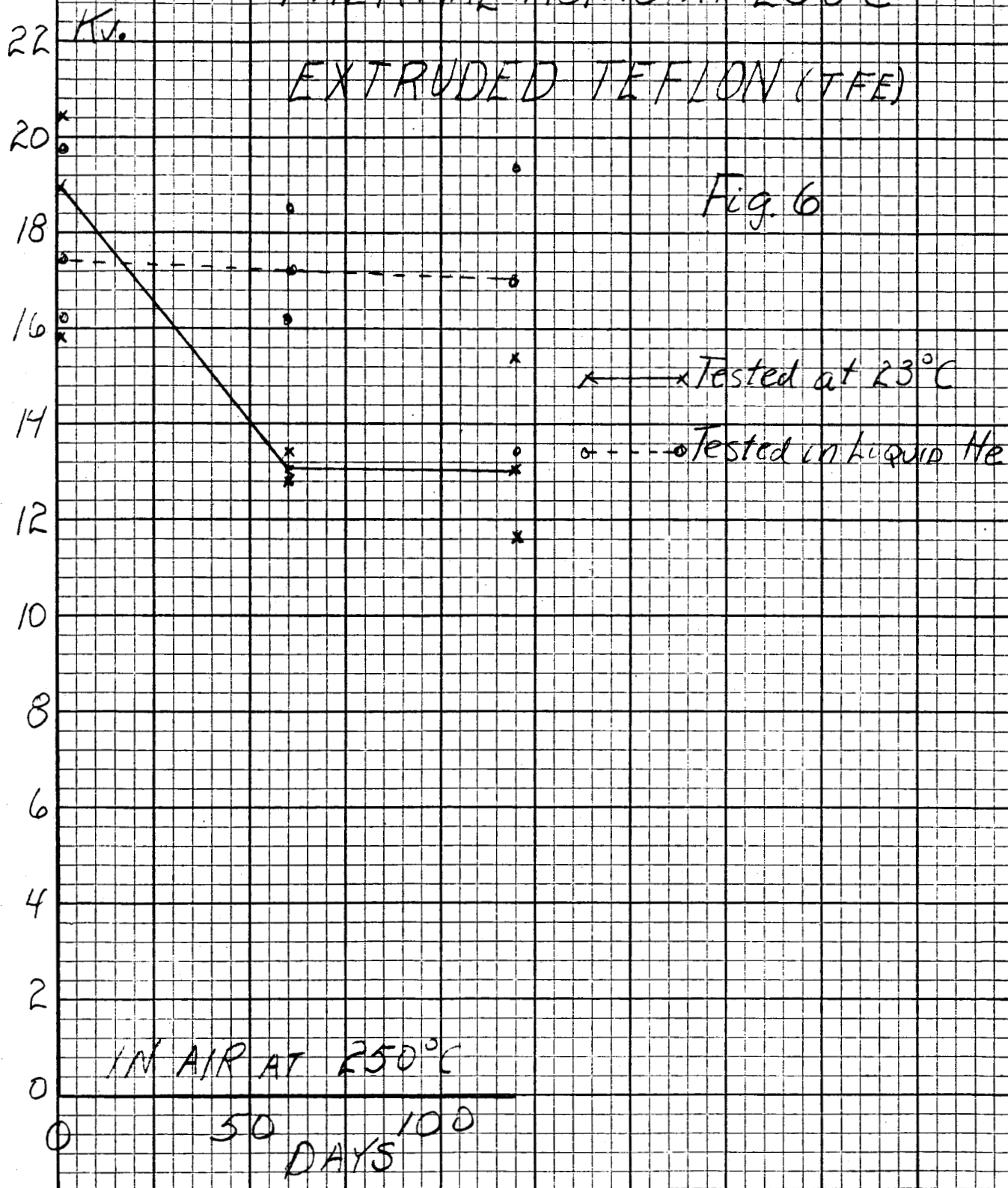


VOLTAGE BREAKDOWN VS. THERMAL AGING AT 120°C EXTRUDED TEFLON (TFE)



VOLTAGE BREAKDOWN VS. THERMAL AGING AT 250°C EXTRUDED TEFLON (TFE)

Fig. 6

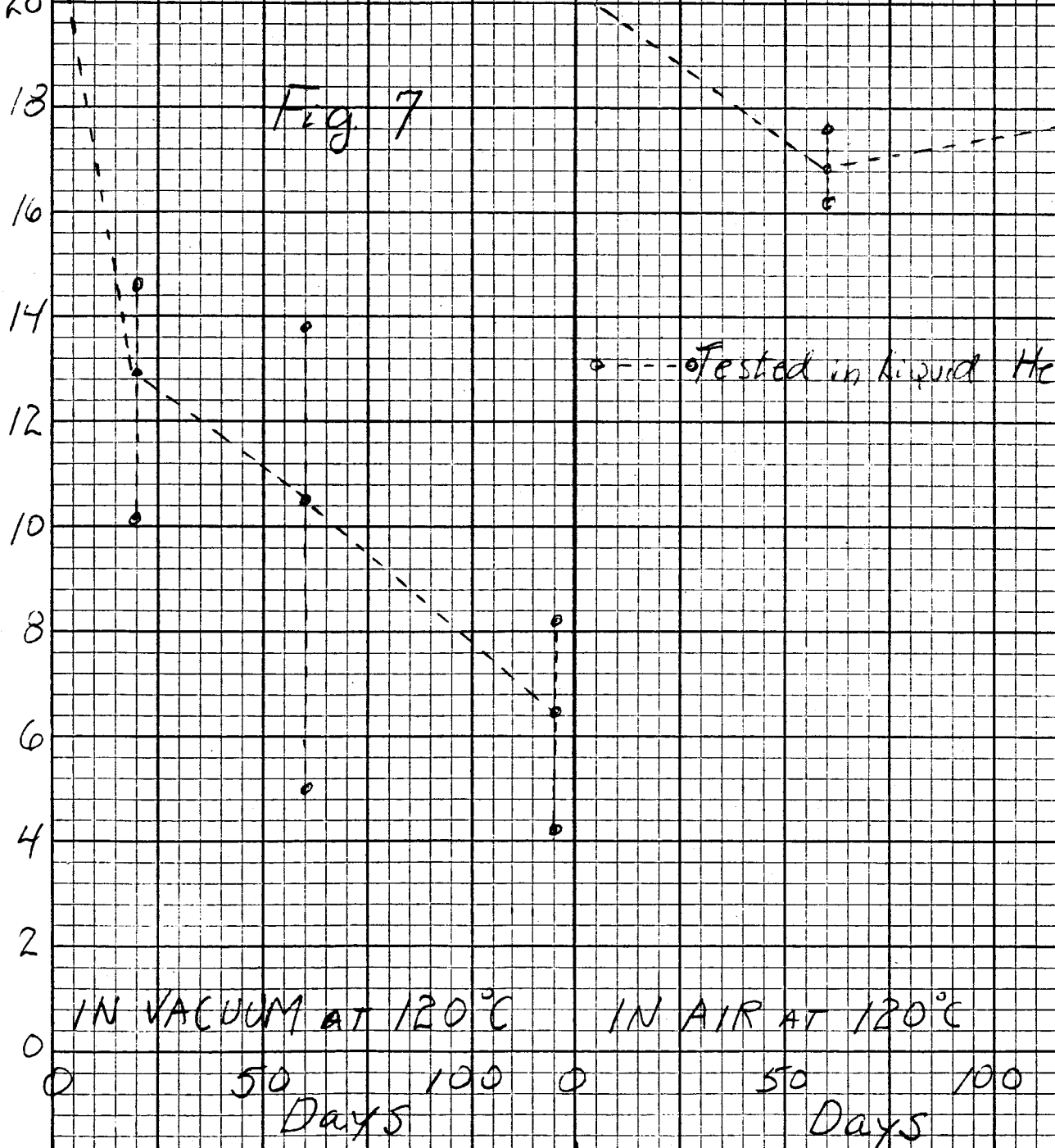


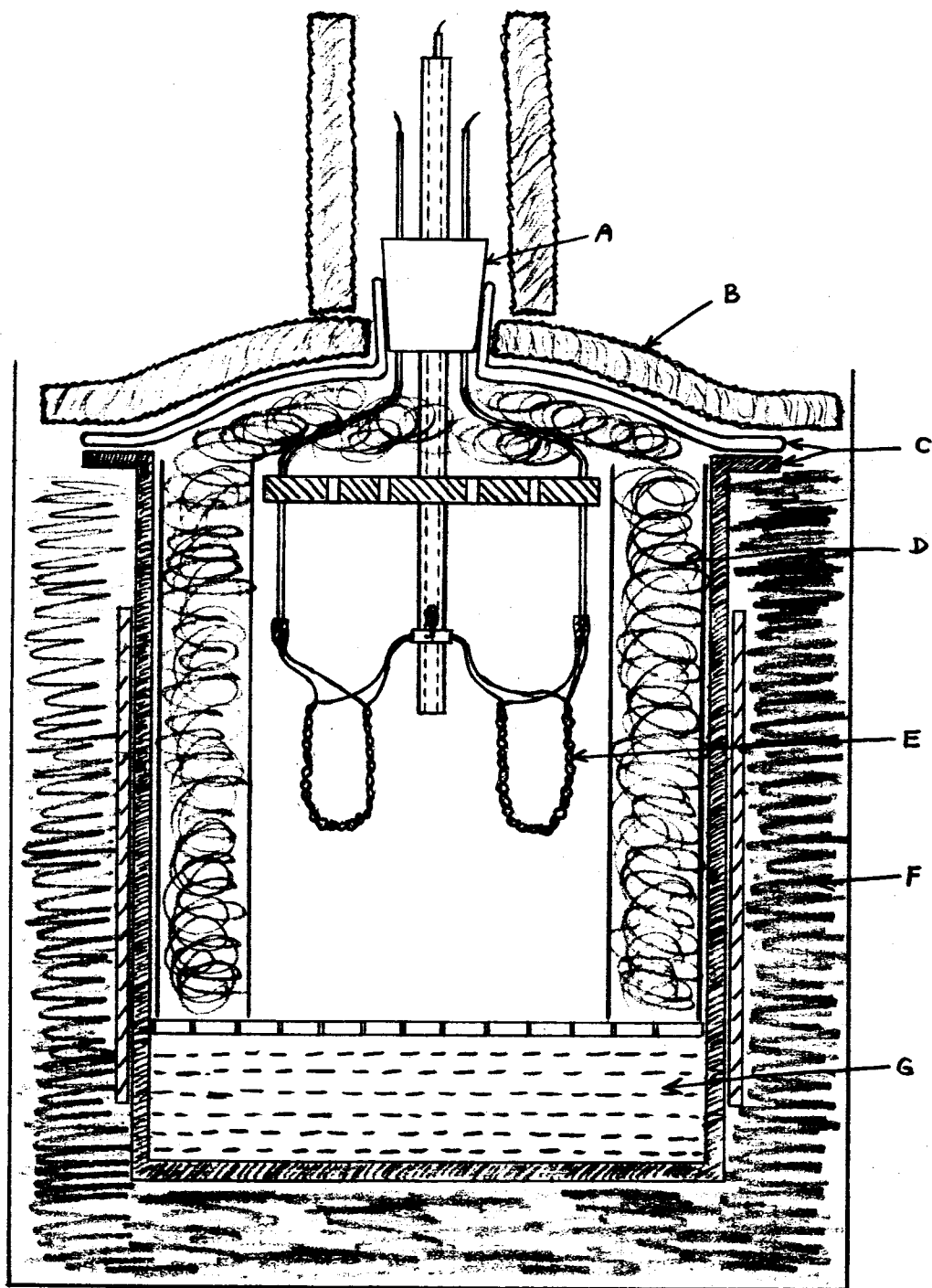
VOLTAGE BREAKDOWN VS. THERMAL AGING AT 120°C

EXTRUDED POLYVINYL CHLORIDE

* * * All Breakdowns at Room Temp are Above 20.5 Kv.

Fig 7

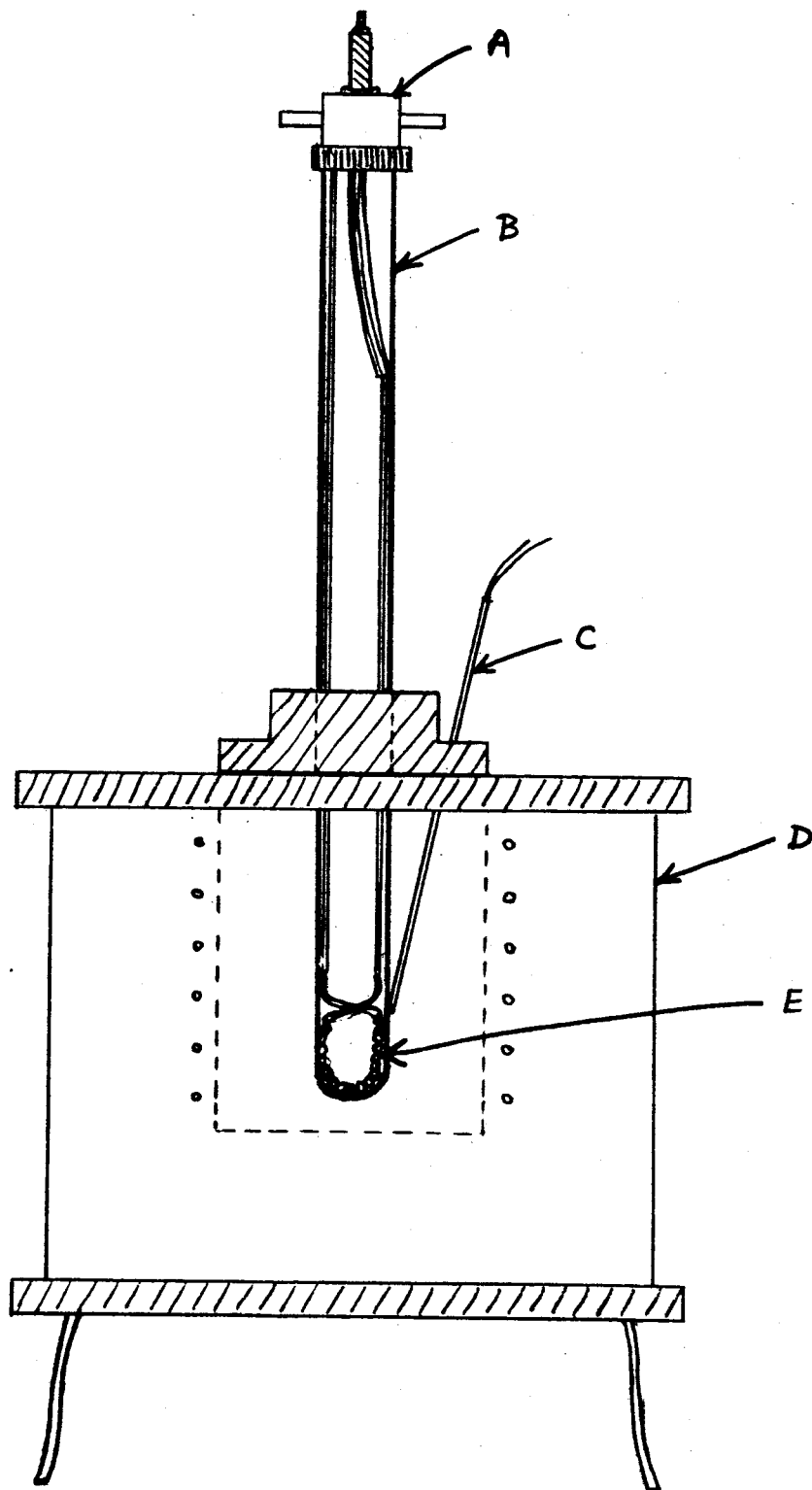




Sketch #1

Humidity Aging, Electric Breakdown Chamber for Twisted Pairs

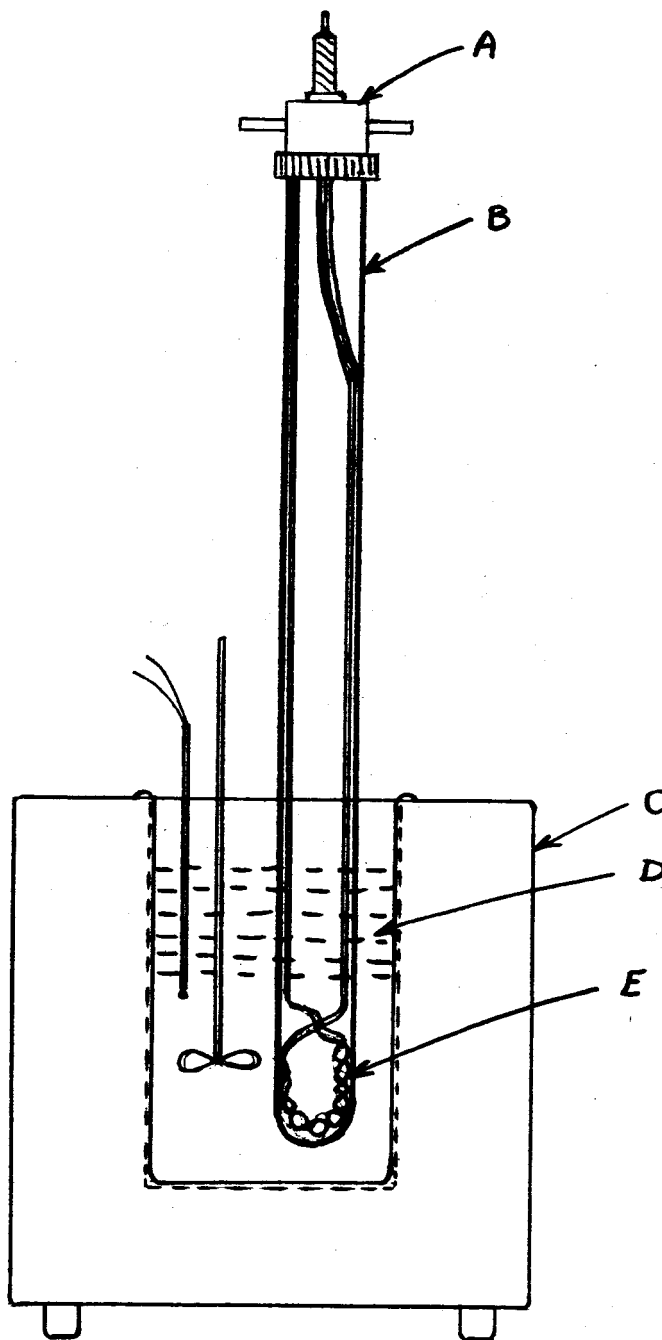
- A. Rubber stopper with 12 high voltage lead wires (Teflon insulated) and a central ground wire in a textolite support tube.
- B. Felt insulation to protect glass cover from outside thermal changes.
- C. Iron tank with glass desiccator cover. Tank heated with stabilized voltage to produce a constant temperature.
- D. Glass wool insulation between two Mylar cylinders to further stabilize the temperature in the chamber.
- E. Test specimens (12) of twisted pairs folded into a U shape to eliminate exposed ends of wire and soldered to the ground ring and high voltage leads.
- F. Rock-wool insulation around the iron tank and strip heaters.
- G. Water-glycerin solution adjusted to produce a 95% relative humidity at 80 C.



Sketch #2

High Temperature (250 C) Vacuum Electric Breakdown Apparatus for Twisted Pairs

- A. Brass compression fitting cap ("O" ring) with high voltage terminal and tubulations for vacuum.
- B. 1-3/8" alumina specimen tube.
- C. Thermocouple to measure the temperature of the tube surface.
- D. Hoskins electric furnace heated with stabilized voltage to produce a constant temperature.
- E. Specimen folded into a U and touching the inside surface of the tube.



Sketch #3

Electric Breakdown Apparatus for -60 C Tests of Twisted Pairs

- A. Brass compression fitting cap ("O" ring) with high voltage terminal and tubulations for dry gas to prevent frost formation.
- B. 1-3/8" alumina specimen tube.
- C. Insulated bath for cooling liquid.
- D. Dry ice-acetone mixture adjusted to -60 C.
- E. Specimen folded into a U and touching the sides of the tube.

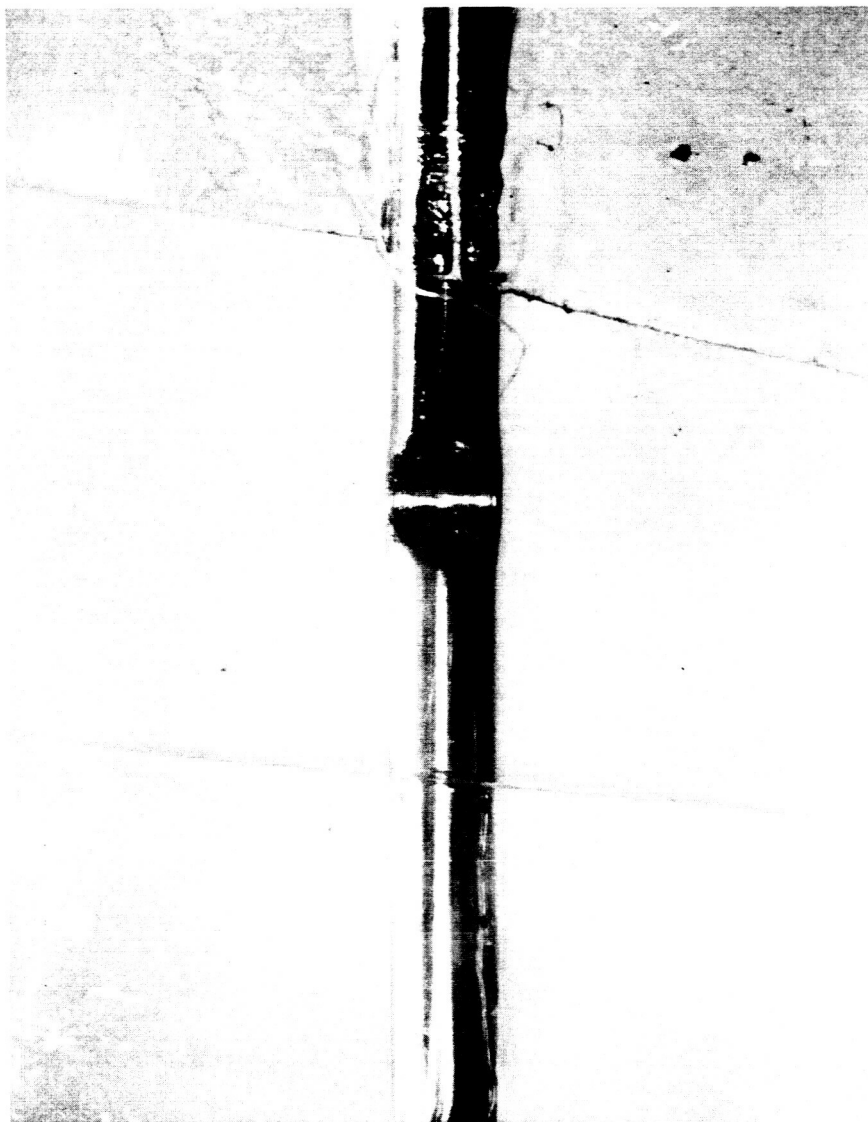


Photo #1

Compression Test of ML Wire in Liquid Nitrogen